

Specification Amendments

Amend paragraphs in the Specification as shown in marked form:

Page 1, first paragraph:

B1 This is a divisional of Application No. 09/759,803 filed January 12, 2001, which is a divisional of Application No. 08/920,419, filed ~~August~~ August 29, 1997.

Page 5, lines 14 – 19:

B2 The present invention also provides a non-diamond-like organic coating on a substrate comprising an organic material comprising at least one major component, wherein the coating has a density that is at least about 50% greater than the density of the major component of the organic material prior to coating. For a one component layer, the non-diamond-like organic coating preferably has substantially the same composition and structure as that of the starting material.

Page 6, insert the following paragraph between lines 15 and 16:

B3 Fig. 5a is a cross-sectional view of the hollow cathode point-source of Fig. 5.

Page 9, line 20 through page 10, line 2:

B4 Using the methods described herein, certain physical and chemical properties of the starting materials are generally maintained. That is, properties of the starting materials, such as coefficient of friction, surface energy, and transparency do not change significantly upon preparing coatings using the methods described herein, as opposed to conventional plasma processes. Thus, the methods of the present invention are very different from conventional plasma processes because the molecules are not significantly broken down to low molecular weight, reactive, species with the methods of the present invention. For example, it is believed that the -Si-O-Si-O- chain of a silicon oil remains substantially ~~in tact~~ intact in the jet plasma process of the present invention.

Page 21, line 14 through page 22, line 24:

B5 The jet plasma vapor deposition apparatus 300 of Fig. 4 also includes a hollow cathode system 315, which includes a point source cathode 316, a feed gas source 317 and a

carrier gas source 318, for generating a plasma, an oil delivery system 320, attached to a valve system 321, and an anode system 322 (e.g., an anode wire as described herein). In this arrangement, the oil delivery system 320 and attached valve system 321 are optional. In the specific embodiment shown in Fig. 4, an imaginary horizontal plane can be drawn from the center of the radio frequency bias electrode 310 to the slot opening of the optional oil delivery system 320, dividing the noncovered surface area (i.e., the deposition area 314) in half. The point source cathode 316 is placed above the imaginary plane and the anode system 322 is placed below the imaginary plane. Plasma extends as a point source from the point source cathode 316 into the vacuum in a cone shape configuration concentrating near the radio frequency bias electrode 310 and at the anode wire 322. Although Fig. 4 is not ~~too~~ to scale, in one embodiment of this system, the point source cathode 316 is placed about 7.5 cm above the imaginary plane and about 7.5 cm away from the surface of the radio frequency bias electrode 310. It is tilted from its horizontal position by about 60° to ensure a downward expansion of the plasma toward the anode wire 322 and the deposition area. The anode wire 322 is placed about 17.5 cm below the imaginary plane and about 5 cm away from the radio frequency bias electrode 310. The dark space ground shield 312 prevents the anode wire 322 from being in-line-of-sight with the deposition area. These distances, lengths, angles, and other dimensions are presented as exemplary only. They are not intended to be limiting.

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Referring to Fig. 5, a point source cathode 400 is shown, which enables the generation of a plasma from a small orifice 403 of a hollowed cylinder 402, which is surrounded by a magnet 408, preferably a circular magnet, and equipped with an electrode, such as the spherical-H.V. electrode 410. The cathode 400 preferably includes a water-cooled cylinder 402, which is typically made of copper, although it can be made of graphite or other electrically and thermally conductive metals. A tube 404, preferably having a circular cross section, is inserted inside a bore 406 of the cylinder 402 having the leading edge 405 recessed within the bore 406 of the cylinder 402 such that it is in the plane of the center line of a circular magnet 408 that surrounds the cylinder 402 at its outlet end. The tube 404 is preferably ceramic, although it can be made of other materials that withstand high temperatures and are electrical insulators. The external surfaces of the cylinder 402 can be shielded with quartz 412 (as by the use of a quartz sleeve) to avoid plasma arcing. This arrangement can be better seen in Fig. 5A 5a, which is a cross section of the point source cathode 400 taken along line A-A, which also shows a water inlet 417 and water outlet 418.

Page 24, lines 7 – 17:

B6 The composition of the coatings can be controlled by means of the concentration and composition of the feed gas passed through the hollow cathode, and the organic material vaporized in the evaporator. The ~~density~~ densities of the coatings are controlled by means of the chamber pressure, the electrical power (current and voltage) supplied by the DC and radio frequency power supplies. The conditions for the formation of high density coatings are generally chosen to balance the bias power to the concentration of the starting material. That is, the specific power density includes bias power density, reaction time, and concentration of starting material. Generally, the specific power density is increased by higher power density and longer reaction time, and decreased by increased concentration of the starting material. Generally, the higher the power density, the ~~more dense~~ denser the coating.

Page 25, line 24 through page 26, line 6:

B7 As stated previously, the plasma is created from a carrier gas or a mixture of a carrier gas and a feed gas. This is referred to herein as the "plasma gas." The carrier gas flow rate can be about 50 standard cubic centimeters per minute (sccm) to about 500 sccm, preferably about 50 sccm to about 100 sccm, and the feed gas flow rate can be about 100 sccm to about 60,000 sccm, preferably about 300 sccm to about 2000 sccm. For example, for carbon deposition rates of about 20 X/second to about 800 X/second, the feed gas flow rate is about 50 sccm to about 350 sccm and the carrier gas flow rate is about 50 sccm to about 100 sccm, with higher feed gas flow rates in combination with lower carrier gas flow rates (typically resulting in higher deposition rates). Generally, for harder coatings, the carrier gas flow rate is increased and the feed gas flow rate is decreased.

Page 27, lines 3 – 10:

B8 The ~~thickness~~ thicknesses of coatings produced by the method of the present invention are typically greater than about 5 nanometers (nm), preferably about 10 nm to about 1000 nm, however, thicker coatings are possible, but not typically needed. The substrate moves through the plasma at a rate designed to provide a coating of a desired thickness. Referring to Fig. 1, the speed at which the substrate 75 travels from roll 76 to roll 78 can be about 10 mm/second to about 4000 mm/second, but is typically about 10

B8f mm/second to about 1500 mm/second for the gas flow rates and pressures and the apparatus described above.

Page 28, line 22 through page 29, line 2:

39 ~~Density was measured by the floating method. Powdered samples were suspended in~~
liquids of varying density and the movement of the suspended particles ~~were~~ was observed.
Upward movement indicated that the particles were less dense than the liquid; downward
movement indicated that the particles were more dense than the liquid. No movement
indicated identical densities. Final readings were made after twelve hours when the particles
usually had risen to the top of the liquid or settled at the bottom. Using liquids with
incremental differences in density, the density of the particles could be bracketed. The
liquids with varying densities used are listed in Table 1.

Page 31, lines 14 - 18:

B10 The anode was similar to that shown in Fig. 4 of U.S. Patent No. 5,464,667 (Köhler et al.) except the enclosing glass box was omitted. Two tungsten wires each 0.1 cm diameter and 40 cm long functioned as anodes that reached a temperature of 800-1100°C with 10-20 amperes of electric current sustained from the plasma. The ~~midsection~~ midsections of the tungsten wires were covered with quartz tubing.

Page 45, line 11 through page 46, line 7:

B11 ~~Samples A, B, C, D, and E were examined by wide angle x-ray scattering (WAXS)~~
for purposes of identifying the presence of crystallinity. Data were collected using a Philips
vertical diffractometer, copper K α radiation, and proportional detector registry of the
scattered radiation. An interference peak on the order of 7.2 Å was produced by all materials
and is the only structural feature observed. The position of the interference maximum
produced by the oil did not change position upon polymerization. This indicates that the
structural features present in the oil maintained their approximate arrangement after
undergoing polymerization. The observed peak was sufficiently broad that the materials
were not considered to possess crystallinity, but rather possessed a structural feature that
repeated itself on a 7 Å length scale. Amorphous carbon and amorphous silica, often used as

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barrier coatings, produce peaks at considerably higher angle, normally between 20 and 30 degrees (2 θ), which correspond to distances on the order of 4.5-3 X. These data indicated that the polymerized ~~material~~ materials were distinctly different from amorphous carbon and silica materials. A different structural feature was obtained from Sample E, which showed a broad peak at 8.7 Angstroms.

Page 49, lines 17 - 25:

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The adhesion of the multi-layer coatings of Examples 6-8 ~~were~~ was evaluated by 90° peel strength testing and summarized in Table 9. In all cases delamination occurred at the interface between the coating and the adhesive tape. In particular, the high peel strength values obtained with samples of Example 8 indicated that the adhesion of the fully polymerized dimethyl silicone layer to the carbon layer and also the adhesion of the carbon layer to the PET film substrate were at least 5.5 N/dm or greater. The high adhesion and the intrinsic low surface energy values of the silicone coatings suggested their use for release coatings and other low surface energy coatings.

Page 51, line 5 through page 52, line 7:

B13
Homogeneous coatings were prepared by a procedure utilizing two feed sources. This method provided the means to obtain new coating properties. Apparatus arrangement and process conditions were similar to those described in Example 3. The hollow cathode slot and the evaporator slot ~~was~~ were placed parallel and in proximity of the radio frequency bias electrode (less than 7 cm). A divider was omitted. A 2.5×10^{-3} cm thick and 15 cm wide type 100H "KAPTON" film obtained from DuPont ~~type 100H~~ was used as the film substrate ~~that and~~ and was transported in loop form around the two rolls of the web drive and the radio frequency bias electrode for multiple deposition passes. The "KAPTON" film also accommodated silicon wafers. After the main vacuum chamber had been evacuated to a pressure of about 1 mTorr, 100 sccm argon was introduced into the argon plasma chamber, i.e., the first compartment of the hollow cathode slot system. The plasma was sustained at about minus 475 volts and a pulsating DC current of about 500 mA. At a flow rate of 150 sccm, acetylene was introduced into the mixing chamber, i.e., the second compartment of the hollow cathode slot system. The hollow cathode slot was powered by a second pulsating DC power supply. The plasma current was 1 amp at about minus 100 volts. The radio frequency

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bias electrode was cooled to about 5-10° C. The bias voltage was minus 1500 volts. The dimethylsilicone oil was introduced into the oil evaporator by way of a microsyringe pump with a feed of 0.05-0.5 ml/minute. A 25 gauge syringe needle was used. The run was completed after 20 passes. The coating was about 2800Å thick and showed excellent water vapor barrier values of 0.17 g/(m²·day). The contact angle and the static coefficient of friction were 99° and 0.22, respectively. The Auger depth profile showed a uniform composition throughout the coating including carbon, silicon, and oxygen.
